

10/657,157

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SDIs in Caplus  
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	ENTRY	SESSION
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FILE COVERS 1907 - 28 Jul 2004 VOL 141 ISS 5  
FILE LAST UPDATED: 27 Jul 2004 (20040727/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s (meth)acrylic esters or meth-acrylic ester or meth acrylic ester  
or(meth)acrylic acid or meth-acrylic acid meth acrylic acid  
MISSING OPERATOR METH)ACRYLIC  
The search profile that was entered contains terms or  
nested terms that are not separated by a logical operator.

=> s (meth)acrylic esters or meth-acrylic ester or meth acrylic ester  
or(meth)acrylic acid or meth-acrylic acid or meth acrylic acid  
MISSING OPERATOR METH)ACRYLIC  
The search profile that was entered contains terms or  
nested terms that are not separated by a logical operator.

```
=> s meth acrylic ester
      33548 METH
        3 METHS
      33550 METH
        (METH OR METHS)
      233395 ACRYLIC
        1256 ACRYLICS
      233754 ACRYLIC
        (ACRYLIC OR ACRYLICS)
      547111 ESTER
      408573 ESTERS
      763869 ESTER
        (ESTER OR ESTERS)
L1      517 METH ACRYLIC ESTER
        (METH(W) ACRYLIC(W) ESTER)
```

```
=> s meth acrylic acid
      33548 METH
        3 METHS
      33550 METH
        (METH OR METHS)
      233395 ACRYLIC
        1256 ACRYLICS
      233754 ACRYLIC
        (ACRYLIC OR ACRYLICS)
      3846720 ACID
      1439656 ACIDS
      4315045 ACID
        (ACID OR ACIDS)
```

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L2            6529 METH ACRYLIC ACID  
                 (METH(W) ACRYLIC (W) ACID)

```
=> s meth-acrylic acid
    33548 METH
      3 METHS
    33550 METH
      (METH OR METHS)
    233395 ACRYLIC
      1256 ACRYLICS
    233754 ACRYLIC
      (ACRYLIC OR ACRYLICS)
    3846720 ACID
    1439656 ACIDS
    4315045 ACID
      (ACID OR ACIDS)
```

L3            6529 METH-ACRYLIC ACID  
                 (METH(W) ACRYLIC (W) ACID)

```
=> s (meth)acrylic acid
MISSING OPERATOR METH) ACRYLIC
The search profile that was entered contains terms or
nested terms that are not separated by a logical operator.
```

```
=> dup rem l1 l2
PROCESSING COMPLETED FOR L1
PROCESSING IS APPROXIMATELY 16% COMPLETE FOR L2
PROCESSING IS APPROXIMATELY 35% COMPLETE FOR L2
PROCESSING IS APPROXIMATELY 54% COMPLETE FOR L2
PROCESSING IS APPROXIMATELY 73% COMPLETE FOR L2
PROCESSING IS APPROXIMATELY 93% COMPLETE FOR L2
PROCESSING COMPLETED FOR L2
L4            6958 DUP REM L1 L2 (88 DUPLICATES REMOVED)
```

```
=> s l4 and (esterif? or transesterif?)
L5            514 S L4
L6            6444 S L4
             121695 ESTERIF?
             19336 TRANSESTERIF?
L7            745 (L5 OR L6) AND (ESTERIF? OR TRANSESTERIF?)
```

```
=> s l7 and alcohol
    214408 ALCOHOL
    147720 ALCOHOLS
    335134 ALCOHOL
      (ALCOHOL OR ALCOHOLS)
    538090 ALC
    179251 ALCS
    629729 ALC
      (ALC OR ALCS)
    748695 ALCOHOL
      (ALCOHOL OR ALC)
L8            244 L7 AND ALCOHOL
```

```
=> s l4 and (process or prepar? or make or made or synthes?)
L9            514 S L4
L10           6444 S L4
             1958444 PROCESS
             1299692 PROCESSES
             2911574 PROCESS
```

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```

        (PROCESS OR PROCESSES)
1480947 PREPAR?
110746 PREP
1954 PREPS
112505 PREP
        (PREP OR PREPS)
1874342 PREPD
21 PREPDS
1874357 PREPD
        (PREPD OR PREPDS)
97386 PREPG
12 PREPGS
97397 PREPG
        (PREPG OR PREPGS)
2490259 PREPN
195592 PREPNS
2638614 PREPN
        (PREPN OR PREPNS)
4367118 PREPAR?
        (PREPAR? OR PREP OR PREPD OR PREPG OR PREPN)
192678 MAKE
148039 MAKES
331508 MAKE
        (MAKE OR MAKES)
1103437 MADE
24 MADES
1103457 MADE
        (MADE OR MADES)
1381870 SYNTHES?
L11 4198 (L9 OR L10) AND (PROCESS OR PREPAR? OR MAKE OR MADE OR SYNTHES?)

=> s l11 and (esterif? or transesterif?)
121695 ESTERIF?
19336 TRANSESTERIF?
L12 594 L11 AND (ESTERIF? OR TRANSESTERIF?)

=> s l12 and alcohol
214408 ALCOHOL
147720 ALCOHOLS
335134 ALCOHOL
        (ALCOHOL OR ALCOHOLS)
538090 ALC
179251 ALCS
629729 ALC
        (ALC OR ALCS)
748695 ALCOHOL
        (ALCOHOL OR ALC)
L13 193 L12 AND ALCOHOL

=> s l13 and inhibitor
447426 INHIBITOR
466871 INHIBITORS
720148 INHIBITOR
        (INHIBITOR OR INHIBITORS)
L14 52 L13 AND INHIBITOR

=> s l13 and polymerization inhibitor
309594 POLYMERIZATION
3945 POLYMERIZATIONS
310136 POLYMERIZATION
```

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(POLYMERIZATION OR POLYMERIZATIONS)  
302253 POLYMN  
7687 POLYMNS  
303220 POLYMN  
(POLYMN OR POLYMNS)  
418088 POLYMERIZATION  
(POLYMERIZATION OR POLYMN)  
447426 INHIBITOR  
466871 INHIBITORS  
720148 INHIBITOR  
(INHIBITOR OR INHIBITORS)  
7284 POLYMERIZATION INHIBITOR  
(POLYMERIZATION(W) INHIBITOR)  
L15 50 L13 AND POLYMERIZATION INHIBITOR

=> d dup rem l14 l15  
L15 IS NOT VALID HERE  
For an explanation, enter "HELP DISPLAY".

=> dup rem l14 l15  
PROCESSING COMPLETED FOR L14  
PROCESSING COMPLETED FOR L15  
L16 52 DUP REM L14 L15 (50 DUPLICATES REMOVED)

=> d l16 ibib hitstr abs 1-52

L16 ANSWER 1 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 1  
ACCESSION NUMBER: 2004:139077 CAPLUS  
DOCUMENT NUMBER: 140:181967  
TITLE: **Process** for manufacturing (**meth**)  
**acrylic esters**  
INVENTOR(S): Hayashi, Katsunori; Kobayashi, Akihiro; Arashima,  
Katsuyasu  
PATENT ASSIGNEE(S): Hitachi Chemical Co., Ltd., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2004051546	A2	20040219	JP 2002-211205	20020719

PRIORITY APPLN. INFO.: JP 2002-211205 20020719

AB In the **process** for manufacturing acrylic ester or methacrylic ester by reacting an **alc.** with acrylic acid or methacrylic acid in a solvent in the presence of an acid catalyst, the reaction mixture is neutralized (upon completion of the **esterification**), washed with water, and a basic lithium salt (at 0.01 to 5 weight% relative to the ester produced) is added to the neutralized liquid, the reaction solvent is distilled off while mol. oxygen is introduced, and the insol. matter is removed by filtration. Thus, tris(2-hydroxyethyl) isocyanurate acrylate (I), showing APHA 40 and good solubility in MeOH and toluene, was **prepared** in 95% yield, vs. I (**prepared** by a reference **process**) showing APHA 40 with good solubility in methanol and low solubility in toluene.

L16 ANSWER 2 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 2  
ACCESSION NUMBER: 2004:159023 CAPLUS  
DOCUMENT NUMBER: 140:199903

TITLE: Esterification process and catalysts for the production of (meth)acrylate esters from (meth)acrylic acid and alcohols

INVENTOR(S): Beste, York Alexander; Stein, Bernd

PATENT ASSIGNEE(S): BASF A.-G., Germany

SOURCE: Ger. Offen., 11 pp.  
CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10317435	A1	20040226	DE 2003-10317435	20030415

PRIORITY APPLN. INFO.: DE 2003-10317435 20030415

AB (meth)acrylate esters (e.g., Bu acrylate) are prepared by the heterogeneous-catalyzed (e.g., Amberlyst 15) conversion of (meth)acrylic acids with at least one alc. in at least one reactor, in which: (A) the water content in the bottom of the azeotropic distillation column is >0.15 ppm; and/or (B) the content of the (meth)acrylic acid in the sump of the azeotrope column is ≤60%; and/or (C) the heterogeneous catalyst before the reaction is contacted with a polymerization inhibitor-containing alc. solution (e.g., 1-butanol and phenothiazine).

L16 ANSWER 3 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 3

ACCESSION NUMBER: 2003:610404 CAPLUS

DOCUMENT NUMBER: 139:149334

TITLE: Process for preparation of (meth)acrylic acid esters

INVENTOR(S): Yada, Shuhei; Goriki, Masayasu; Nakamura, Mitsuo

PATENT ASSIGNEE(S): Mitsubishi Chemical Corporation, Japan

SOURCE: PCT Int. Appl., 11 pp.  
CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003064370	A1	20030807	WO 2003-JP594	20030123

W: AE, AG, AL, AU, BA, BB, BR, BZ, CA, CN, CO, CR, CU, DM, DZ, EC, GD, GE, HR, HU, ID, IL, IN, IS, KP, KR, LC, LK, LR, LT, LV, MA, MG, MK, MN, MX, NO, NZ, OM, PH, PL, RO, SC, SG, TN, TT, UA, US, UZ, VC, VN, YU, ZA, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

JP 2003292472	A2	20031015	JP 2003-13454	20030122
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PRIORITY APPLN. INFO.: JP 2002-21374 A 20020130

AB This invention pertains to a method for producing (meth)acrylic acid esters which comprises feeding (meth)acrylic acid and an alc. to a reactor, wherein a polymerization inhibitor is dissolved in the reaction mixture (Meth)acrylic acid and (meth)acrylic acid esters are prevented from

polymerizing on the member present in the gas-phase part of the reactor, and a continuous operation can be conducted over long. For example, acrylic acid was reacted with BuOH in the presence of p-toluenesulfonic acid and hydroquinone to give Bu acrylate.

REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 4 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 4

ACCESSION NUMBER: 2003:396830 CAPLUS

DOCUMENT NUMBER: 138:385915

TITLE: Method for producing (meth)acrylic acid esters of polyhydric alcohols

INVENTOR(S): Martin, Friedrich-Georg; Wartini, Alexander; Dernbach, Matthias; Schroeder, Juergen; Sirch, Tilman

PATENT ASSIGNEE(S): BASF Aktiengesellschaft, Germany

SOURCE: PCT Int. Appl., 41 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 6

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003042151	A1	20030522	WO 2002-EP12491	20021108
WO 2003042151	C1	20040624		
W:				
AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW:				
GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
DE 10156116	A1	20030626	DE 2001-10156116	20011115

PRIORITY APPLN. INFO.: DE 2001-10156116 A 20011115

OTHER SOURCE(S): MARPAT 138:385915

AB (Meth)acrylic acid esters of polyhydric alcs. are manufactured by reacting (meth)acrylic acid and the corresponding polyhydric alcs. in the presence of  $\geq 1$  acid catalyst and, optionally,  $\geq 1$  polymerization inhibitor and a solvent, whereby the polyhydric alc. contains  $< 500$  ppm HCHO. Thus, trimethylolpropane containing 282 ppm acetal-bound HCHO was esterified with acrylic acid in cyclohexane mixture containing p-MeOC<sub>6</sub>H<sub>4</sub>OH, H<sub>3</sub>PO<sub>2</sub>, CuCl<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> to give product having d. 1.1041 g/cm<sup>3</sup> and dynamic viscosity 85 mPa·s (23°), vs. d. 1.1153 g/cm<sup>3</sup> and dynamic viscosity 246 mPa·s for similar product prepared by use of trimethylolpropane containing 1400 ppm of acetal-bound HCHO.

REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 5 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 5

ACCESSION NUMBER: 2003:166982 CAPLUS

DOCUMENT NUMBER: 138:188258

TITLE: Preparation of 2-phenylethyl (meth)acrylate

INVENTOR(S): Doi, Junichi; Sonobe, Hiroshi; Matsumoto, Satoshi

10/657,157

PATENT ASSIGNEE(S): Mitsubishi Rayon Co., Ltd., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2003064024	A2	20030305	JP 2001-254623	20010824
PRIORITY APPLN. INFO.:			JP 2001-254623	20010824

AB The compound is prepared by esterification of (meth)acrylic acid with 2-phenylethyl alc. in the presence of 0.05-0.5 mol equivalent (based on 1 mol alc.) of acid catalysts. Methacrylic acid was esterified with 2-phenylethyl alc. in the presence of p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H, N,N'-di-2-naphthyl-p-phenylenediamine, and p-methoxyphenol at 72-83° for 10 h, washed with alkalies, and distilled to give 95% 2-phenylethyl methacrylate with 99.7% purity.

L16 ANSWER 6 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2003:35382 CAPLUS

DOCUMENT NUMBER: 138:73685

TITLE: Process for preparing alkyl (meth)acrylates

INVENTOR(S): Venter, Jeremia Jesaja; Mirabelli, Mario Giuseppe Luciano

PATENT ASSIGNEE(S): Rohm and Haas Company, USA

SOURCE: U.S., 7 pp., Cont.-in-part of U.S. Ser. No. 192,675, abandoned.

CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6506930	B1	20030114	US 1999-417400	19991013
PRIORITY APPLN. INFO.:			US 1997-66939P	P 19971117
			US 1998-192675	B2 19981116

AB The process provides for the synthesis of alkyl (meth)acrylates, hydrolysis of process impurities into starting materials and separation of starting materials and reaction products in one reactor. A typical process comprises: (A) charging a reactor with a C1-4 alc.; a (meth)acrylic acid; a strong acid catalyst selected from the group consisting of sulfuric acid, alkylsulfonic acid and polymer supported alkylsulfonic acid; at least one inhibitor selected from the group consisting of 2,2,6,6-tetramethyl-1-piperidinyloxy, 4-hydroxy-2,2,6,6-tetramethyl-1-piperidinyloxy, 4-methacryloyloxy-2,2,6,6-tetramethyl piperidinyloxy free radical and 4-hydroxy-2,2,6,6-tetramethyl N-hydroxypiperidine; and at least 5% water to form a reaction mixture; (B) reacting the reaction mixture to form a C1-4 alkyl (meth)acrylate and process impurities, wherein the process impurities are hydrolyzed in said reactor; and (C) separating the C1-4 alkyl (meth)acrylate and water formed during the reaction from the reaction mixture

REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT



L16 ANSWER 7 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 6

ACCESSION NUMBER: 2002:845303 CAPLUS  
 DOCUMENT NUMBER: 137:338384  
 TITLE: **Esterification process** for the  
 production of (meth)acrylate esters  
 INVENTOR(S): Nestler, Gerhard; Geisendoerfer, Matthias  
 PATENT ASSIGNEE(S): BASF AG, Germany  
 SOURCE: Ger. Offen., 12 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10154714	A1	20021107	DE 2001-10154714	20011109
DE 10246869	A1	20030327	DE 2002-10246869	20021008

PRIORITY APPLN. INFO.: DE 2001-10154714 A1 20011109

AB The production of (meth)acrylate esters (e.g., 2-ethylhexyl acrylate) by is achieved by acid-catalyzed **esterification** of (meth) **acrylic acids** (e.g., acrylic acid) with the appropriate **alc.** (e.g., 2-ethylhexanol) in a homogeneous liquid phase in the presence of a polymerization **inhibitor** (e.g., phenothiazine) and/or an **inhibitor** mixture, one accomplishes the **esterification** (stage 1) in a reaction zone, which is equipped with at least one distillation unit, over which one separates the reaction water as well as olefins, **alc.**, acetic acid esters and propionic acid ester, formed with the **esterification**, condensed and in an aqueous and an organic phase are separated, the discharge from the reaction zone from stage 1 is lead into a catalyst separation stage (stage 2) and into a **esterification** -catalyzed bottoms product and the (meth)acrylate esters head product is separated, from this (meth)acrylate ester-containing head product in a following stage the remaining (meth)acrylate ester-containing stream/current is separated into a light-boiling fraction (stage 4) and the (meth)acrylate ester essentially freed of acetic acid ester and output **alc.** recycled, from the released acetic acid ester and output **alc.** the (meth)acrylate ester **made** from stage 4 in a pure distillation (stage 6) separates from the high-boiling solvents and the high-boiling solvent-containing stream is subjected to a thermal and/or catalytic treatment.

L16 ANSWER 8 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 7

ACCESSION NUMBER: 2002:403632 CAPLUS  
 DOCUMENT NUMBER: 136:402195  
 TITLE: **Transesterification process** for  
 the production of higher alkyl (meth)acrylate esters  
 from lower-alkyl (meth)acrylate esters  
 INVENTOR(S): Nestler, Gerhard; Rauh, Ulrich; Schroeder, Juergen  
 PATENT ASSIGNEE(S): BASF AG, Germany  
 SOURCE: Ger. Offen., 12 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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 DE 10127941 A1 20020529 DE 2001-10127941 20010608  
 WO 2002100815 A1 20021219 WO 2002-EP5821 20020528  
 W: CN, JP, US  
 RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,  
 PT, SE, TR  
 EP 1399409 A1 20040324 EP 2002-747325 20020528  
 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,  
 IE, FI, CY, TR

PRIORITY APPLN. INFO.: DE 2001-10127941 A 20010608  
 WO 2002-EP5821 W 20020528

OTHER SOURCE(S): MARPAT 136:402195

AB (meth)acrylate esters [e.g., 2-(dimethylamino)ethyl acrylate] are prepared in high yield and selectivity by the transesterification of lower-alkyl (meth)acrylate esters (e.g., Bu acrylate) with a higher alc. [e.g., 2-(dimethylamino)ethanol] in the presence of a polymerization inhibitor (e.g., phenothiazine and hydroquinone monomethyl ether) and a transesterification catalyst (e.g., tetra-Bu titanate) or a catalyst mixture, and where one separates the free byproduct lower alkanol (e.g., 1-butanol) and at least partly supplies the production of the lower-alkyl (meth)acrylate by acidification of the process waste water and esterification of the residual (meth)acrylic acid (e.g., acrylic acid) with the byproduct alkanol.

L16 ANSWER 9 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 8

ACCESSION NUMBER: 2002:462390 CAPLUS

DOCUMENT NUMBER: 137:33676

TITLE: Procedure for manufacture of (meth) acrylic acid esters

INVENTOR(S): Martin, Friedrich-Georg; Nestler, Gerhard; Schroeder, Juergen

PATENT ASSIGNEE(S): BASF AG, Germany

SOURCE: Ger. Offen., 4 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10063176	A1	20020620	DE 2000-10063176	20001218
WO 2002050015	A1	20020627	WO 2001-EP14903	20011217
W: US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
EP 1345887	A1	20030924	EP 2001-984858	20011217
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				

US 2004030179 A1 20040212 US 2003-433614 20030617

PRIORITY APPLN. INFO.: DE 2000-10063176 A 20001218  
 WO 2001-EP14903 W 20011217

AB (meth)acrylic acid esters (of mol. weight >200) are obtained by esterification of (meth) acrylic acid with alcs. in the presence of  $\geq 1$  acid catalyst,  $\geq 1$  polymerization inhibitor, and an organic solvent, which forms an azeotrope with water, whereby the mixture is heated to the b.p. in an apparatus with a distillation unit, column and condenser,

the azeotrope is distilled off and the organic solvent is recirculated to the column at least partially contacting a copper-containing material, such as distillation column packings or separation efficient fittings. Thus, acrylic acid

2380, tripropylene glycol 2880, cyclohexane 2300, p-toluenesulfonic acid 120, 50% phosphinic acid 9.4, and hydroquinone monomethylether 4.7 parts were mixed in a 10-L-reactor with a double layer heating and distillation column

(5 + 60 cm). The reaction water formed was distilled off as azeotrope with cyclohexane, whereby after after condensation the organic phase formed was separated and recirculated to the column, which was filled with copper Raschig-rings at the top and glass rings under it. Within 8 h, 546 parts of water was separated, so that a 97% yield of **esterification** took place and no polymerization was observed in the column.

L16 ANSWER 10 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 9

ACCESSION NUMBER: 2002:462389 CAPLUS

DOCUMENT NUMBER: 137:47591

TITLE: Procedure for manufacture of higher (meth)  
**acrylic acid** esters

INVENTOR(S): Martin, Friedrich-Georg; Nestler, Gerhard; Schroeder, Juergen

PATENT ASSIGNEE(S): BASF AG, Germany

SOURCE: Ger. Offen., 6 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 6

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10063175	A1	20020620	DE 2000-10063175	20001218
WO 2002055472	A1	20020718	WO 2001-EP14636	20011213
W: US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
EP 1345886	A1	20030924	EP 2001-984833	20011213
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
WO 2002050014	A1	20020627	WO 2001-EP14902	20011217
W: US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
EP 1343748	A1	20030917	EP 2001-994797	20011217
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
US 2004019235	A1	20040129	US 2003-433612	20030617
US 2004024241	A1	20040205	US 2003-450438	20030618
PRIORITY APPLN. INFO.:				
				DE 2000-10063175 A
				DE 2001-10152680 A
				WO 2001-EP14636 W
				WO 2001-EP14902 W

AB Higher (meth)**acrylic acid** esters (of mol. weight >200) are obtained by **esterification** of (meth)**acrylic acid** with higher **alcs.** in the presence of  $\geq 1$  acid catalyst,  $\geq 1$  polymerization **inhibitor**, and an organic solvent, which forms an azeotrope with water, whereby the the mixture is heated to the b.p. in an apparatus with a distillation unit, column and condenser

and the reactor content is circulated via an external evaporator, preferably an self-circulating evaporator. Thus, (meth) **acrylic acid** 172, methoxyphenol 9.2, phenothiazine 0.3, 50% phosphinic acid 23.8, 65% p-toluenesulfonic acid 497, and cyclohexane 1030 g were mixed in a 10-L-reactor with an external evaporator, distillation column, and condenser. A circulation evaporator (a heat exchanger comprising a nest of boiler tubes filled with oil) was used. After evaporating the water from the reaction mixture, 344 g (meth) **acrylic acid** and 6000 g Me polyethylene glycol were added to give after 330 min 207 g aqueous phase and 6890 g crude ester. The procedure permits the **preparation** of higher (meth) **acrylic acid** esters without use of copper salts as polymerization **inhibitors**.

L16 ANSWER 11 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 10  
 ACCESSION NUMBER: 2001:481835 CAPLUS  
 DOCUMENT NUMBER: 135:61734  
 TITLE: Manufacture of (meth)acrylate esters with recycling polymerization **inhibitor**-containing residues  
 INVENTOR(S): Nakahara, Osamu; Kamioka, Masatoshi  
 PATENT ASSIGNEE(S): Nippon Shokubai Kagaku Kogyo Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001181233	A2	20010703	JP 2000-311413	20001012
US 6649787	B1	20031118	US 2000-684396	20001010
PRIORITY APPLN. INFO.:			JP 1999-289345	A 19991012

AB (meth)acrylate esters are manufactured by **esterification** of (meth) **acrylic acid** with C1-4 aliphatic **alcs.** in the presence of acid catalysts and distillation of the reaction mixts. using an acid separation column., a low boiling substance-separation column, and a rectifying column with returning ..... from the rectifying column to the **esterification** and/or the separation/purification **process**. Thus, (a) acrylic acid containing phenothiazine (I), (b) BuOH, and (c) I-containing high boiling residue collected from the rectifying column were passed through a cation exchanger-packed reactor and the reaction mixture was distilled with using fresh I as a polymerization **inhibitor** in the acid separation and rectifying **processes**. Hydroquinone monomethyl ether was also used as a polymerization **inhibitor** in another rectifying column. and Bu acrylate was collected. The polymerization **inhibitors** were recovered and reused in distillation of the acrylate ester.

L16 ANSWER 12 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 11  
 ACCESSION NUMBER: 2001:150618 CAPLUS  
 DOCUMENT NUMBER: 134:193858  
 TITLE: **Esterification process** for the production of (meth)acrylate esters with the azeotropic distillation of **process** water  
 INVENTOR(S): Leube, Hartmann F.; Leidinger, Kurt; Geisendoerfer, Matthias; Beck, Erich  
 PATENT ASSIGNEE(S): BASF A.-G., Germany  
 SOURCE: Ger. Offen., 6 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent

10/657,157

LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19941136	A1	20010301	DE 1999-19941136	19990830
EP 1081125	A1	20010307	EP 2000-117813	20000818
EP 1081125	B1	20030611		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
AT 242759	E	20030615	AT 2000-117813	20000818
ES 2200763	T3	20040316	ES 2000-117813	20000818
US 6437172	B1	20020820	US 2000-644824	20000824

PRIORITY APPLN. INFO.: DE 1999-19941136 A 19990830

AB (meth)acrylate esters (e.g., the methacrylate ester of propoxylated trimethylolpropane) are **prepared** in high yield and selectivity by the **esterification** of a high-boiling mono- or polyhydric **alc.** (e.g., propoxylated trimethylolpropane) with (meth) **acrylic acid** in the presence of an **esterification** catalyst (e.g., p-toluenesulfonic acid), a polymerization **inhibitor** (e.g., hydroquinone monomethyl ether), and an azeotropic distillation agent, such as (cyclo)aliphatic hydrocarbons (e.g., cyclohexane),  
for the removal of **process** water, which azeotropic distillation agent is dosed into the reaction mixture during the **esterification** reaction for both the removal of **process** water and so as to control the boiling temperature of the reaction.

L16 ANSWER 13 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 12

ACCESSION NUMBER: 2001:114630 CAPLUS

DOCUMENT NUMBER: 134:163471

TITLE: Production of esters of (meth) **acrylic acid** and polyhydric **alcohols** and device therefor

INVENTOR(S): Roessler, Harald; Fies, Matthias; Gutsche, Bernhard; Stalberg, Theo

PATENT ASSIGNEE(S): Cognis Deutschland G.m.b.H., Germany

SOURCE: Ger. Offen., 6 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19937911	A1	20010215	DE 1999-19937911	19990811
WO 2001012315	A1	20010222	WO 2000-EP7496	20000803
W: KR, US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
EP 1204472	A1	20020515	EP 2000-958367	20000803
EP 1204472	B1	20030129		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY				
ES 2191637	T3	20030916	ES 2000-958367	20000803

PRIORITY APPLN. INFO.: DE 1999-19937911 A 19990811  
WO 2000-EP7496 W 20000803

AB Esters based on (meth)acrylic acid and

polyhydric **alcs.** are produced in a reactor whereby the liquid reaction mixture contains polymerization **inhibitors** and whereby one at least partly separates the produced water of reaction in the vapor state, which is characterized by the fact that a part of the gas/vapor phase of the reaction mixture is removed from the reactor, then partially condensed in a dephlegmator and then fed to an ascending gas/steam mixture as well as the condensate coming from the head of the dephlegmator, with a polymerization **inhibitor** being added, and with provisions being **made** for recycling to the reactor. The **process** permits the use of a smaller excess of unsatd. acid, decreases the chance of polymerization, shortens the reaction time, and decreases the organic wastewater content.

L16 ANSWER 14 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 13  
 ACCESSION NUMBER: 2000:600284 CAPLUS  
 DOCUMENT NUMBER: 133:193609  
 TITLE: Radical polymerization **inhibitors** containing phenothiazine adducts and manufacture of (meth)acrylate esters using them  
 INVENTOR(S): Sato, Hiroyuki; Fukumura, Takanori; Ohizumi, Fumitaka  
 PATENT ASSIGNEE(S): Chisso Corp., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2000234002	A2	20000829	JP 1999-36716	19990216
PRIORITY APPLN. INFO.:			JP 1999-36716	19990216
AB The <b>inhibitors</b> comprise adducts of phenothiazine with S-containing acidic compds. and are used for <b>preparation</b> of (meth)acrylate esters from (meth) <b>acrylic acid</b> or (meth)acryloyl chloride and hydroxy compds. Thus, a reaction of methacrylic acid with 1-(2-hydroxyethyl)-2-pyrrolidone at 130° for 4 h in the presence of H2SO4 and phenothiazine gave 2-(2-pyrrolidonyl)ethyl methacrylate.				

L16 ANSWER 15 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 14  
 ACCESSION NUMBER: 2000:412204 CAPLUS  
 DOCUMENT NUMBER: 133:43958  
 TITLE: **Preparation** of high-purity (meth)**acrylic acid** esters  
 INVENTOR(S): Yoshida, Koichi; Tokuda, Masanori; Sonobe, Hiroshi; Ohkita, Motomu  
 PATENT ASSIGNEE(S): Mitsubishi Rayon Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2000169429	A2	20000620	JP 1998-351392	19981210
PRIORITY APPLN. INFO.:			JP 1998-351392	19981210
OTHER SOURCE(S):			MARPAT 133:43958	
GI				

L16 ANSWER 16 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 15  
ACCESSION NUMBER: 2000:374769 CAPLUS  
DOCUMENT NUMBER: 133:8284  
TITLE: Polyoxyalkylene (meth)acrylic  
acid esters and their polymers, their  
manufacture, and use of the polymers as cement  
dispersants  
INVENTOR(S): Hirata, Takeshi; Yuasa, Tsutomu  
PATENT ASSIGNEE(S): Nippon Shokubai Kagaku Kogyo Co., Ltd., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 19 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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JP 2000154247	A2	20000606	JP 1998-328686	19981118
PRIORITY APPLN. INFO.:			JP 1998-328686	19981118

L16 ANSWER 17 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 16  
ACCESSION NUMBER: 2000:277696 CAPLUS  
DOCUMENT NUMBER: 132:279644  
TITLE: **Process** for the **preparation** and

extraction of (meth)acrylate esters of mono- and polyhydric **alcohols** using dense-phase carbon dioxide

INVENTOR(S): Tweedy, Harrel E.  
PATENT ASSIGNEE(S): UCB, S.A., Belg.  
SOURCE: Eur. Pat. Appl., 18 pp.  
CODEN: EPXXDW  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 995738	A1	20000426	EP 1999-120221	19991011
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
JP 2000128829	A2	20000509	JP 1999-296617	19991019
PRIORITY APPLN. INFO.:		US 1998-174412	A	19981019

AB The present invention relates to a **process** for preparing acrylate esters, methacrylate esters, polyester acrylates (e.g., trimethylolpropane triacrylate) and/or polyester methacrylates by reacting acrylic and/or methacrylic acid with a monohydroxy compound and/or a polyhydroxy compound (e.g., trimethylolpropane) in a reaction vessel, in the presence of a catalyst (e.g., Amberlyst A15), a polymerization **inhibitor** (e.g., phenothiazine), and dense-phase carbon dioxide. The dense phase carbon dioxide is in either a sub-critical, near-critical, critical, or supercrit. phys. state. The advantages of using dense-phase carbon dioxide as the solvent include the **preparation** of (meth)acrylate esters and polyester (meth)acrylates in an environmentally friendly manner by reducing or eliminating the need for hydrocarbon solvents, reducing or eliminating the problems associated with handling hydrocarbon solvents, simplifying recovery of the products, and recovery and recycle of reactants as compared to the conventional **esterification synthesis**. In addition, these products have improved color and purity.

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 18 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 17  
ACCESSION NUMBER: 2000:240714 CAPLUS  
DOCUMENT NUMBER: 132:279640  
TITLE: **Preparation** of (meth)acrylate esters and polyester (meth)acrylates in the presence of a catalyst and **inhibitor** under microwave heating

INVENTOR(S): Tweedy, Harrel E.  
PATENT ASSIGNEE(S): UCB, S.A., Belg.  
SOURCE: Eur. Pat. Appl., 15 pp.  
CODEN: EPXXDW

DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 995738	A1	20000412	EP 1999-119616	19991004
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,				



IE, SI, LT, LV, FI, RO  
 US 6175037 B1 20010116 US 1998-168991 19981009  
 JP 2000119216 A2 20000425 JP 1999-289458 19991012  
 PRIORITY APPLN. INFO.: US 1998-168991 A 19981009

AB Acrylic or methacrylic acid is reacted with a monohydroxy- or polyhydroxy-containing compound in the presence of a catalyst and polymerization

**inhibitor** using microwave energy as the heating source, which can be applied to the reaction vessel continuously or intermittently by pulsing. Preferably the monohydroxy-containing compds. are C1-18 linear or branched aliphatic, cycloaliph. and aromatic compds., such as methanol and cyclohexanol; and the polyhydroxy-containing compds. are C2-36 polyols having .apprx.2-10 hydroxy groups, such as cyclohexandiol, butanediol and tripropylene glycol. Advantages of using microwave energy include higher temps. coupled with shorter residence times, reduced production costs, increased capacity, lower energy costs, effective use of raw materials, and solventless processing which is environmentally friendly. Thus, a mixture of 7.3 g methacrylic acid, 5.0 g 1,6-hexanediol, 0.1 g phenothiazine, and 0.6 g methanesulfonic acid (70% aqueous solution) was polymerized

in a microwave oven at 20% power (.apprx.200 W) for 30 s followed by 20 s shaking for 2 cycles, and 18 cycles of 20-s heating/20-s agitation. After a total of 7 min cumulative microwave exposure the sample contained (GC, area % values) 74.2% 1,6-hexanediol dimethacrylate, 22.2% 1,6-hexanediol monomethacrylate, and 1.3% 1,6-hexanediol along with some other minor peaks.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 19 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 18  
 ACCESSION NUMBER: 2000:209660 CAPLUS  
 DOCUMENT NUMBER: 132:237568  
 TITLE: Method for production of polyoxyalkylene acrylates  
 INVENTOR(S): Hirata, Tsuyoshi; Yuasa, Tsutomu  
 PATENT ASSIGNEE(S): Nippon Shokubai Co., Ltd., Japan; Nippon Catalytic Chem. Ind.  
 SOURCE: Eur. Pat. Appl., 36 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 989108	A2	20000329	EP 1999-118597	19990921
EP 989108	A3	20010124		
EP 989108	B1	20031126		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
US 6265495	B1	20010724	US 1999-399491	19990920
KR 2000023420	A	20000425	KR 1999-41117	19990922
JP 2000159882	A2	20000613	JP 1999-269435	19990922
JP 3094024	B2	20001003		
CN 1256265	A	20000614	CN 1999-125143	19990922
CN 1134406	B	20040114		
JP 2000212128	A2	20000802	JP 1999-311664	19991101
JP 3390382	B2	20030324		

PRIORITY APPLN. INFO.: JP 1998-268122 A 19980922  
 JP 1998-328687 A 19981118

AB A method for the production of an **esterified** product is provided which affords an **esterified** product with high quality by repressing, during the **esterification** reaction of an **alc** . with (meth)acrylic acid, the occurrence of impurities, particularly gel, to be formed owing to the polymerization of the **alc.** and the (meth)acrylic acid as raw materials, the **esterified** product consequently formed, or the mixture thereof. Specifically, a method for the production of an **esterified** product of this invention comprises **esterifying** an **alc.** represented by the following formula (1):  $R_1O(R_2O)_nH$  wherein  $R_1$  represents a hydrocarbon group of 1 to 30 carbon atoms,  $R_2O$  represents an oxyalkylene group of 2 to 18 carbon atoms, providing that the repeating units,  $R_2O$ , may be the same or different and that when the  $R_2O$ 's are in the form of a mixture of two or more species, the repeating units,  $R_2O$ , may be added either in a block form or in a random form, and  $n$  represents an average addition mol number of oxyalkylene groups and is in the range of 0 to 300, with (meth)acrylic acid in a dehydrating solvent in the presence of an acid catalyst and a polymerization inhibitor, wherein a reaction temperature during the **esterification** reaction is not higher than 130°C and a circulation speed of the solvent during the **esterification** reaction is not less than 0.5 cycle/h.

L16 ANSWER 20 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 19

ACCESSION NUMBER: 2000:909652 CAPLUS

DOCUMENT NUMBER: 134:72023

TITLE: **Process** for the manufacture of (meth)acrylic acid esters

INVENTOR(S): Paulus, Wolfgang; Reich, Wolfgang; Beck, Erich; Jaworek, Thomas; Koeniger, Rainer

PATENT ASSIGNEE(S): Basf A.-G., Germany

SOURCE: Ger. Offen., 6 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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DE 19929258	A1	20001228	DE 1999-19929258	19990625
US 6458991	B1	20021001	US 2000-592736	20000613
JP 2001026569	A2	20010130	JP 2000-187845	20000622
EP 1063225	A2	20001227	EP 2000-113365	20000623
EP 1063225	A3	20020717		

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO

PRIORITY APPLN. INFO.: DE 1999-19929258 A 19990625

AB Compds. with **alc.** OH groups are **esterified**, in the presence of Cu(II) salts or their mixts. with Cu(I) salts as polymerization inhibitors, with acrylic or methacrylic acid in the presence of a hydrocarbon (b.p. 60-140°) for azeotropic removal of water at 90-150°. After **esterification** the Cu salt is precipitated as CuS and the ester is separated. The resulting light-colored esters, obtained in high yields, are particularly suitable for colored pigmented coatings on substrates such as wood, paper, mineral building materials, plastic or metal.

L16 ANSWER 21 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:34537 CAPLUS  
 DOCUMENT NUMBER: 130:95963  
 TITLE: Zirconium catalyzed **transesterification process** for preparing synthetic wax monomers  
 INVENTOR(S): Schlaefer, Francis William; Gross, Andrew William  
 PATENT ASSIGNEE(S): Rohm and Haas Company, USA  
 SOURCE: U.S., 5 pp.  
 CODEN: USXXAM  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5856611	A	19990105	US 1997-950443	19971015
PRIORITY APPLN. INFO.:			US 1997-950443	19971015

AB A zirconium catalyzed **transesterification process** for preparing synthetic wax monomers comprises reacting a synthetic wax **alc.** with a (meth)acrylic acid ester in the presence of a zirconium catalyst and an **inhibitor**. The monomers are useful in a wide range of polymers.  
 REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 22 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 20  
 ACCESSION NUMBER: 1998:421476 CAPLUS  
 DOCUMENT NUMBER: 129:82071  
 TITLE: Manufacture of tetrahydrobenzyl (meth)acrylate by using polymerization **inhibitors** and oxygen gas for reaction efficiency  
 INVENTOR(S): Fujiwara, Keisuke  
 PATENT ASSIGNEE(S): Daicel Chemical Industries, Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10175919	A2	19980630	JP 1996-353733	19961218
PRIORITY APPLN. INFO.:			JP 1996-353733	19961218

OTHER SOURCE(S): MARPAT 129:82071  
 AB Tetrahydrobenzyl **alc.** (I) is **esterified** with (meth)acrylic acid in the presence of O<sub>2</sub>-containing gases and polymerization **inhibitors** of (1) (A) quinones, hindered phenols, nitrosoamines, and/or phenylenediamines or (B) phenothiazines, RR'NOH (R, R' = H, alkyl, aryl), Cu(S<sub>2</sub>CNR<sub>12</sub>)<sub>2</sub> (R<sub>1</sub> = alkyl, aryl), and/or Fe(S<sub>2</sub>CNR<sub>12</sub>)<sub>3</sub> or (2) (A') hydroquinone, hydroquinone monomethyl ether (II), benzoquinone, 3,5-di-tert-butyl-4-hydroxytoluene (III), N-nitrosodiphenylamine, and/or N,N'-diphenylphenylenediamine or (B') phenothiazine, Cu(S<sub>2</sub>CNMe<sub>2</sub>)<sub>2</sub> (IV), Cu(S<sub>2</sub>CN<sub>Et</sub>)<sub>2</sub>, Cu(S<sub>2</sub>CNPr<sub>2</sub>)<sub>2</sub>, Fe(S<sub>2</sub>CNMe<sub>2</sub>)<sub>3</sub>, and/or Et<sub>2</sub>NOH, and the resulting crude solution is distilled with O<sub>2</sub>-containing gases and the above polymerization **inhibitors** for purification. Alternatively, the **esterification** is carried out by using the gases and the **inhibitors** of A', and the resulting solution is distilled with the gases and the **inhibitors** of B'. Thus, a solution

of I was bubbled with air and reacted with methacrylic acid in the presence of a catalyst and polymerization **inhibitors** of II and III. Then, the product solution after catalyst removal was refluxed with IV to give tetrahydrobenzyl methacrylate with yield 84%.

L16 ANSWER 23 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 21  
 ACCESSION NUMBER: 1998:421475 CAPLUS  
 DOCUMENT NUMBER: 129:68146  
 TITLE: Production of tetrahydrobenzyl (meta)acrylate in simple condition for improved reaction efficiency  
 INVENTOR(S): Fujiwara, Keisuke  
 PATENT ASSIGNEE(S): Daicel Chemical Industries, Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10175918	A2	19980630	JP 1996-353732	19961218
PRIORITY APPLN. INFO.:			JP 1996-353732	19961218
OTHER SOURCE(S): MARPAT 129:68146				
AB Tetrahydrobenzyl <b>alc.</b> (I) is <b>esterified</b> with (meth)acrylic acid in the presence of polymerization <b>inhibitors</b> and catalysts in dehydration solvents of C <sub>≤10</sub> hydrocarbons to carry out dehydration by azeotropic distillation Catalysts are removed from the resulting crude solution to attain catalyst concentration ≤10%, and then the crude solution is distilled for purification Thus, I in n-hexane was reacted with methacrylic acid in the presence of methanesulfonic acid (catalyst) and polymerization <b>inhibitors</b> . The product solution was heated, stirred with water, and separated to organic and aqueous layers, so that 98.3% (based on initial catalyst content) of the catalyst was removed. Then, the resulting solution was distilled with another polymerization <b>inhibitor</b> to give tetrahydrobenzyl methacrylate with yield 84%.				

L16 ANSWER 24 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 22  
 ACCESSION NUMBER: 1998:126650 CAPLUS  
 DOCUMENT NUMBER: 128:193033  
 TITLE: Preparation of (meth)acrylate esters  
 INVENTOR(S): Okada, Shinji; Muraue, Takao; Mishina, Hiroya  
 PATENT ASSIGNEE(S): Toa Gosei Chemical Industry Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10053559	A2	19980224	JP 1996-227883	19960809
PRIORITY APPLN. INFO.:			JP 1996-227883	19960809
AB Title esters are <b>prepared</b> by reacting (meth)acrylic acids and <b>alcs.</b> in the presence of catalysts with removing H <sub>2</sub> O by distillation, in which aqueous basic solns. are added				

to the distillate for adjusting pH at  $\geq 6.5$ . Corrosion on the reactors caused by the distillates or polymerization of the (meth) **acrylic acid** in the distillates are avoided in the **process**. Thus, acrylic acid, BuOH, H<sub>2</sub>SO<sub>4</sub>, hexane, and polymerization **inhibitor** were continuously charged at the rates of 100, 115, 1.4, 56, and 0.3 part/h, resp., at 120° into a reactor equipped with (a) a distillation column connected with a condenser and with (b) a separator from which the organic layer is recycled to a distillation column. Crude ester and

H2O

were continuously discharged from the reactor bottom and from the separator, resp., with continuously charging 25%-NaOH into the vapor from the distillation column in order to maintain pH at 7-8 at the separator. The reaction could be continued for 100 days with no polymer formation in the separator and corrosion of the SUS-304 condenser.

L16 ANSWER 25 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 23

ACCESSION NUMBER: 1998:165487 CAPLUS

DOCUMENT NUMBER: 128:193025

TITLE: Manufacture of halogenated (meth) **acrylic esters** and poly (meth) acrylates

INVENTOR(S): Hofstraat, Johannes Willem; Wakselman, Claude; Lequesne, Christelle; Wiersum, Ulfert Elle; Blazejewski, Jean Claude

PATENT ASSIGNEE(S): Akzo Nobel N.V., Neth.

SOURCE: Eur. Pat. Appl., 21 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 824096	A2	19980218	EP 1997-202331	19970729
EP 824096	A3	19980408		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
CA 2212733	AA	19980213	CA 1997-2212733	19970811
JP 10095751	A2	19980414	JP 1997-230482	19970813
PRIORITY APPLN. INFO.:			EP 1996-202276	19960813

OTHER SOURCE(S): MARPAT 128:193025

AB The present invention is directed to the **preparation** of (meth)acrylates of halogenated **alcs.** by direct **esterification** of the **alcs.** with (meth)acryloyl chloride wherein at least one 2,6-substituted pyridine derivative is used as a polymerization

**inhibitor** at a temperature  $< 50^\circ$ . Suitable 2,6-substituted pyridine derivs. are 2,6-lutidine, 2,4,6-collidine or 2,6-di-tert-butyl-4-methylpyridine. With the halogenated (meth)acrylate esters **prepd** according to the invention, very pure homo- and copolymers can be **prepared**, resulting in polymers having a low optical loss of  $\leq 0.1$  dB/cm at 1300 nm and  $< 0.4$  dB/cm at 1550 nm. The polymers are useful in making a waveguide.

L16 ANSWER 26 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 24

ACCESSION NUMBER: 1998:808209 CAPLUS

DOCUMENT NUMBER: 130:223626

TITLE: Study on catalytic properties and polymerization retardation of heteropoly acid in the

preparation of acrylate  
 AUTHOR(S): Ma, Liqun; Yang, Yulin  
 CORPORATE SOURCE: Department of Chemical Engineering, Industry  
 Institute, Qiqihar University, Qiqihar, 161006, Peop.  
 Rep. China  
 SOURCE: Huagong Shikan (1998), 12(10), 16-18  
 CODEN: HUSHFT; ISSN: 1002-154X  
 PUBLISHER: Huagong Shikan Zazhishe  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Chinese

AB Ethylene glycol diacrylate, tetrahydrofurfuryl methacrylate and octadecyl  
 methacrylate were prepared by esterification in the  
 presence of heteropoly acid catalyst. Suitable heteropoly acid-  
 alc. ratio was 1.0-1.5% and suitable (meth)  
 acrylic acid-alc. was 2.2:apprx.2.4:1. The  
 heteropoly acid showed good polymerization inhibition effect for Me  
 methacrylate.

L16 ANSWER 27 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 25

ACCESSION NUMBER: 1997:754319 CAPLUS

DOCUMENT NUMBER: 128:23268

TITLE: Preparation of (meth)  
 acrylic acid esters with  
 polymerization prevention and apparatus therefor  
 INVENTOR(S): Okata, Shinji; Muraue, Takao; Mishina, Hiroya  
 PATENT ASSIGNEE(S): Toa Gosei Chemical Industry Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.  
 CODEN: JKXXAF

DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 09301921	A2	19971125	JP 1996-140966	19960510
PRIORITY APPLN. INFO.:			JP 1996-140966	19960510

AB (Meth)acrylic acids are esterified  
 with alcs. in the presence of catalysts to give crude products,  
 which are added with polymerization inhibitors and Michael addition  
 products of (meth)acrylic acid esters, and  
 remove low-b.p. impurities by distillation The Michael addition products are  
 added  
 so that their concentration in resulting residue liqs. being 3-30%. Thus,  
 heating 100 parts acrylic acid with 152 parts 2-ethylhexyl alc.  
 at 120° in the presence of H<sub>2</sub>SO<sub>4</sub> and phenothiazine (I) in PhMe gave  
 a crude ester liquid, which was purified by distillation at 135-145° in the  
 presence of I, 2-ethylhexyl β-2-ethylhexyloxypropionate, and  
 2-ethylhexyl β-acryloxypropionate to give 2-ethylhexyl acrylate in  
 high yield.

L16 ANSWER 28 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 26

ACCESSION NUMBER: 1995:737585 CAPLUS

DOCUMENT NUMBER: 123:143297

TITLE: Process for the transesterification  
 of (meth)acrylic acid  
 esters.

INVENTOR(S): Knebel, Joachim; Pfirrmann, Martina; Ohl, Thomas  
 PATENT ASSIGNEE(S): Rohm GmbH, Germany  
 SOURCE: Eur. Pat. Appl., 10 pp.

CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 663386	A1	19950719	EP 1995-100110	19950105
EP 663386	B1	19990414		
R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE				
DE 4401132	A1	19950720	DE 1994-4401132	19940117
AT 178880	E	19990415	AT 1995-100110	19950105
CA 2140304	AA	19950718	CA 1995-2140304	19950116
			DE 1994-4401132	A 19940117

PRIORITY APPLN. INFO.:

OTHER SOURCE(S): CASREACT 123:143297; MARPAT 123:143297

AB Acrylate and methacrylate esters are **prepared** in higher yield and purity by **transesterification** of lower alkyl (meth)acrylates  $\text{CH}_2:\text{C}(\text{R}_1)\text{CO}_2\text{R}_2$  [ $\text{R}_1 = \text{H}, \text{Me}$ ;  $\text{R}_2 = \text{C}_1\text{-6 alkyl}$ ] with mono- or polyhydric **alcs.** in the presence of mixed catalysts consisting of 5-95% diorganyltin oxide  $\text{R}_3\text{R}_4\text{Sn}:\text{O}$  [ $\text{R}_3, \text{R}_4 = \text{C}_1\text{-12 aliphatic, aromatic, or araliph. group}$ ] and 95-5% organotin halide  $\text{R}_3\text{R}_4\text{SnX}_2$  or  $\text{R}_3\text{SnX}_3$  [ $\text{X} = \text{Cl}, \text{Br}, \text{iodo, cyano, isocyanato, isothiocyanato}$ ]. For example, **transesterification** of triethylene glycol with excess Me methacrylate in the presence of equal amts. of  $\text{Bu}_2\text{Sn}:\text{O}$  and  $\text{Bu}_2\text{SnCl}_2$ , plus 4-HOC<sub>6</sub>H<sub>4</sub>OMe (polymerization **inhibitor**), under reflux with distillation of MeOH and Me methacrylate, gave triethylene glycol dimethacrylate in 87% yield, and containing only 0.8% monomethacrylate byproduct and only 1.3% Me methacrylate. In contrast, a run with only  $\text{Bu}_2\text{SnCl}_2$  catalyst gave no reaction. Similarly **prepared** in good yields were ethylene glycol dimethacrylate, pentaerythritol tetramethacrylate and tetraacrylate, and tetrahydrofurfuryl methacrylate.

L16 ANSWER 29 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 27

ACCESSION NUMBER: 1995:207908 CAPLUS

DOCUMENT NUMBER: 122:82299

TITLE: Manufacture of (meth)acrylic acid esters

INVENTOR(S): Ri, Shotaku; Takahashi, Katsuji; Okuda, Ryuji

PATENT ASSIGNEE(S): Dainippon Ink &amp; Chemicals, Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06234700	A2	19940823	JP 1993-24124	19930212

PRIORITY APPLN. INFO.:

JP 1993-24124 19930212

AB The compds. are manufactured by **esterification** of (meth)acrylic acid with **alcs.** in the presence of polymerization **inhibitors** and **esterification** catalysts,  $\geq 1$  of which being water-soluble; and washing the reaction mixture with H<sub>2</sub>O to remove the water-soluble polymerization **inhibitors** and/or catalysts. Thus, heating acrylic acid with trimethylolpropane in the presence of 2-methyl-1-phenol-4-sulfonic acid, Cu sulfate, and p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H in PhMe at 65° and 130 torr for 10 h, and washing the reaction mixture with H<sub>2</sub>O gave 99.5% acrylic esters with 100% removal of the

polymerization **inhibitors** and the catalyst.

L16 ANSWER 30 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 28

ACCESSION NUMBER: 1995:207907 CAPLUS  
 DOCUMENT NUMBER: 122:82298  
 TITLE: Manufacture of (meth)**acrylic acid esters**  
 INVENTOR(S): Ri, Shotaku; Takahashi, Katsuji; Okuda, Ryuji  
 PATENT ASSIGNEE(S): Dainippon Ink & Chemicals, Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06234699	A2	19940823	JP 1993-24125	19930212
PRIORITY APPLN. INFO.:			JP 1993-24125	19930212

AB The compds. are manufactured by (trans)**esterification** of (meth)**acrylic acid esters** with **alcs.** in the presence of water-soluble polymerization **inhibitors** and water-soluble catalysts, washing with H<sub>2</sub>O, and recycling of the polymerization **inhibitors** and the catalysts. Thus, acrylic acid was heated with trimethylolpropane in the presence of 2-methyl-1-phenol-4-sulfonic acid, Cu sulfate, and p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H in PhMe at 65° and 130 torr for 10 h, and washed with H<sub>2</sub>O twice to give 99.5% acrylic acid ester. The polymerization **inhibitors** and the catalyst in the 1st washing water were recycled and used for the reaction giving 99.5% acrylate.

L16 ANSWER 31 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 29

ACCESSION NUMBER: 1994:410191 CAPLUS  
 DOCUMENT NUMBER: 121:10191  
 TITLE: Manufacture of (meth)acrylates  
 INVENTOR(S): Takahashi, Katsuji; Tani, Juichiro; Ri, Shotaku; Okuda, Tatsushi  
 PATENT ASSIGNEE(S): Dainippon Ink & Chemicals, Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 13 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06016594	A2	19940125	JP 1993-55628	19930316
JP 2914075	B2	19990628		
PRIORITY APPLN. INFO.:			JP 1992-111414	19920430

AB (Meth)acrylates are **synthesized** by **esterification** of (meth)**acrylic acids** with **alcs.** or by **transesterification** of (meth)acrylates with **alcs.** in the presence of a compound which acts as both an **esterification** catalyst and a polymerization **inhibitor**. Compds. which can be used for this purpose are aromatic sulfonic acids with phenolic hydroxy, amino, nitro or nitroso groups and/or partially transition metal ion-exchanged strongly acidic cation-exchange resins. For example, 201 g trimethylolpropane and 422 g acrylic acid reacted in the presence of 12.5 g hydroquinone-2-sulfonic acid in cyclohexane-toluene with 98.6% **alc.** conversion



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and 95.6% ester yield; no polymeric species was detected.

L16 ANSWER 32 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 30

ACCESSION NUMBER: 1994:410190 CAPLUS

DOCUMENT NUMBER: 121:10190

TITLE: **Preparation of (meth)**

**acrylic acid esters**

INVENTOR(S): Tani, Juichiro; Okuda, Ryuji; Takahashi, Katsuji; Ri, Shotaku

PATENT ASSIGNEE(S): Dainippon Ink & Chemicals, Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 19 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06009496	A2	19940118	JP 1993-55627	19930316

PRIORITY APPLN. INFO.: JP 1992-111415 19920430

AB The title compds. are easily **prepared** by reacting **(meth) acrylic acid** with **alcs.** in the presence of water-soluble **esterification** catalysts and water-soluble polymerization **inhibitors**, then washing the products with water to remove the catalysts and **inhibitors**. The uses of these catalysts and polymerization **inhibitors** simplifies the workup **process**. Thus, heating trimethylolpropane 201, acrylic acid 422, Na hydroquinonesulfonate 3.1, and p-toluenesulfonic acid 12.5 g in 13 g PhMe and 112 g cyclohexane at 100° for 6 h and working up gave a corresponding ester at 95.8% yield.

L16 ANSWER 33 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 31

ACCESSION NUMBER: 1994:165213 CAPLUS

DOCUMENT NUMBER: 120:165213

TITLE: **Preparation of acrylic ester or methacrylic ester**

INVENTOR(S): Haga, Masami

PATENT ASSIGNEE(S): Idemitsu Petrochemical Co, Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05320095	A2	19931203	JP 1992-128363	19920521

PRIORITY APPLN. INFO.: JP 1992-128363 19920521

AB The title esters without discoloration are **prepared** by treating (**meth) acrylic acid** with **alcs.** in the presence of acid catalysts and aromatic compds. having  $\geq 1$  alkyl and  $\geq 2$  OH (per 1 benzene ring) as polymerization **inhibitors**. Thus, 1.20 mol acrylic acid was **esterified** with 1.00 mol 2-ethylhexyl **alc.** (I) in cyclohexane in the presence of 50 mg 2-tert-butylhydroquinone and 0.50 g concentrated H<sub>2</sub>SO<sub>4</sub> while bubbling with N having O content 6 volume% at 84-98° for 6 h to give an ester with ASTM hue L value 0.5 without polymer generation at 98.9 mol% I conversion.

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L16 ANSWER 34 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 32

ACCESSION NUMBER: 1993:473881 CAPLUS

DOCUMENT NUMBER: 119:73881

TITLE: Curable (meth)acrylate compositions with low viscosity and manufacture thereof

INVENTOR(S): Yagi, Hirobumi; Sugimura, Toshiro

PATENT ASSIGNEE(S): Nippon Shokubai Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 04353503	A2	19921208	JP 1991-129715	19910531
JP 3107851	B2	20001113		
JP 2000103815	A2	20000411	JP 1999-205291	19910531

PRIORITY APPLN. INFO.: JP 1991-129715 A3 19910531

AB The title composition usable with high filler content and in photocurable epoxy resist inks contain inorg. materials and (meth)acrylates **prepared** from (meth)**acrylic acid** and/or (meth)acrylates and **alcs.** in the presence of a Cu compound A composition from 80 g trimethylolpropane triacrylate **prepared** from trimethylolpropane and acrylic acid in the presence of p-toluenesulfonic acid and Cu<sub>2</sub>O and 20 g talc had viscosity (25°) 900 cP, compared with 1700 cP for a control using hydroquinone polymerization **inhibitor** in place of Cu<sub>2</sub>O.

L16 ANSWER 35 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 33

ACCESSION NUMBER: 1994:580428 CAPLUS

DOCUMENT NUMBER: 121:180428

TITLE: **Process for preparing** fluorohaloalkyl (meth)acrylates

INVENTOR(S): Chvatal, Zdenek; Dedek, Vaclav; Mazac, Jiri; Marousek, Vladimir; Bednar, Bohumil

PATENT ASSIGNEE(S): Vysoka Skola Chemicko-Technologicka, Czech.

SOURCE: Czech., 4 pp.

CODEN: CZXXA9

DOCUMENT TYPE: Patent

LANGUAGE: Czech

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 276012	B6	19920318	CS 1989-2377	19890417

PRIORITY APPLN. INFO.: CS 1989-2377 19890417

OTHER SOURCE(S): MARPAT 121:180428

AB CH<sub>2</sub>:CRCO<sub>2</sub>(CH<sub>2</sub>)<sub>n</sub>X (I; R = H, Me; X = BrCF<sub>2</sub>CF<sub>2</sub>, ClCF<sub>2</sub>CF<sub>2</sub>, BrCF<sub>2</sub>CFCl, ClCF<sub>2</sub>CFCl, 1-chlorotrifluoroethyl, BrCF<sub>2</sub>CF<sub>2</sub>CF<sub>3</sub>, F<sub>3</sub>CCFBrCF<sub>2</sub>, F<sub>3</sub>CCBrCF<sub>3</sub>; n = 1-4) were **prepared** by acid-catalyzed **esterification** of the appropriate **alc.** with (meth)**acrylic acid** in the presence of polymerization **inhibitor** or by **esterification** of the acid chloride in an alkaline medium in the presence of a phase-transfer catalyst. For example, 6 g CH<sub>2</sub>:CMeCOCl was added dropwise at 0° to a stirred mixture of 10 g BrCF<sub>2</sub>CF<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH, 3 g KOH, 0.32 g Bu<sub>4</sub>NBr, 50 mL CH<sub>2</sub>Cl<sub>2</sub>, and 50 mL H<sub>2</sub>O and the whole stirred for 2.5 h to give 8.5 g of 97%-pure (GLC) I (R = Me, X = BrCF<sub>2</sub>CF<sub>2</sub>, n = 2).

L16 ANSWER 36 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 34  
 ACCESSION NUMBER: 1991:409589 CAPLUS  
 DOCUMENT NUMBER: 115:9589  
 TITLE: Manufacture of (meth)acrylate esters  
 INVENTOR(S): Haga, Masami  
 PATENT ASSIGNEE(S): Idemitsu Petrochemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 03034956	A2	19910214	JP 1989-166565	19890630
JP 07064791	B4	19950712		

PRIORITY APPLN. INFO.: JP 1989-166565 19890630

AB In **preparation** of (meth)acrylate esters by reaction of (meth) **acrylic acid** with **alcs.** in solvents in the presence of acid catalysts, byproduct formation is reduced by (A) using (meth) **acrylic acids** containing polymerization **inhibitors**, (B) carrying out the reaction under inert gas containing 0.1-10 volume% O, and optionally (C) treating the reaction mixts. with aqueous alkali at 40-95° and washing the separated oily phase with H2O at 40-95°. Thus, stirring a mixture of 186.3 g n-dodecyl **alc** ., 90.4 g methacrylic acid, 0.5 g concentrate H2SO4, 50 mg methoxyhydroquinone, and 80 mL PhMe under N containing 6.0 volume% O at 125-151°, mixing the reaction mixture with aqueous NaOH at 60° for 5 min, separating the oily phase, washing with H2O at 60° for 5 min, and distillation of the residue gave 98.7 mol% n-dodecyl methacrylate of 98.9% purity.

L16 ANSWER 37 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 35  
 ACCESSION NUMBER: 1991:7402 CAPLUS  
 DOCUMENT NUMBER: 114:7402  
 TITLE: Manufacture of (meth)acrylate esters of polyhydric **alcohols** having reduced color  
 INVENTOR(S): Ritter, Wolfgang; Sitz, Hans Dieter; Speitkamp, Ludwig  
 PATENT ASSIGNEE(S): Henkel K.-G.a.A., Germany  
 SOURCE: Ger. Offen., 6 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3843938	A1	19900628	DE 1988-3843938	19881224
EP 376091	A1	19900704	EP 1989-123220	19891215
R: GR				
WO 9007487	A1	19900712	WO 1989-EP1550	19891215
W: JP, US				
RW: AT, BE, CH, DE, ES, FR, GB, IT, LU, NL, SE				
EP 449912	A1	19911009	EP 1990-900819	19891215
EP 449912	B1	19940302		
R: BE, CH, DE, ES, FR, GB, IT, LI, NL				
JP 04502468	T2	19920507	JP 1990-501716	19891215
CA 2006434	AA	19900624	CA 1989-2006434	19891221

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US 5198574 A 19930330 US 1991-720445 19910624  
PRIORITY APPLN. INFO.: DE 1988-3843938 19881224  
WO 1989-EP1550 19891215

AB The title monomers are **prepared** by the **esterification** of polyhydric **alcs.** with (meth)acrylic **acids** in the presence of nonsubstituted phenol polymerization **inhibitors**, **transesterification** catalysts, and activated charcoal color reducing agent. Thus, acrylic acid 928.8, propoxylated neopentyl glycol (OH value 416 mg KOH/g) 1560.4, p-toluene sulfonic acid 87.1, activated charcoal 124.5 and hydroquinone (1100 ppm based on product mixture) 2.5 g were mixed together under a air/N mixture (5 volume% O; 20 L/h) at 135-143° for 5 h, then the activated carbon was removed by filtration, producing a crude product having acid value 34 mg KOH/g, OH value 10 mg KOH/g, Gardner color value <1, viscosity 92 mPa-s, which was mixed with 4 L of aqueous 16% NaCl and 4% NaHO3 solution in the presence of 200 ppm hydroquinone monomethyl ether and dried at 80°/40 mBar for 3 h, and filtered, producing a product having acid value <1 mg KOH/g, OH value <15 mg KOH/g and Gardner color value 3, vs. <1, <15, and 8-9 for a control product **prepared** without the addition of activated charcoal.

L16 ANSWER 38 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 36

ACCESSION NUMBER: 1991:7401 CAPLUS

DOCUMENT NUMBER: 114:7401

TITLE: Solventless manufacture of (meth)acrylate esters of polyhydric **alcohols**

INVENTOR(S): Ritter, Wolfgang; Sitz, Hans Dieter; Speitkamp, Ludwig

PATENT ASSIGNEE(S): Henkel K.-G.a.A., Germany

SOURCE: Ger. Offen., 8 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3843930	A1	19900628	DE 1988-3843930	19881224
EP 376090	A1	19900704	EP 1989-123219	19891215
R: GR				
WO 9007485	A1	19900712	WO 1989-EP1548	19891215
W: JP, US				
RW: AT, BE, CH, DE, ES, FR, GB, IT, LU, NL, SE				
EP 449913	A1	19911009	EP 1990-900821	19891215
EP 449913	B1	19940309		
R: BE, CH, DE, ES, FR, GB, IT, LI, NL				
JP 04502466	T2	19920507	JP 1990-501714	19891215
JP 3036832	B2	20000424		
ES 2050427	T3	19940516	ES 1990-900821	19891215
CA 2006431	AA	19900624	CA 1989-2006431	19891221
US 5648518	A	19970715	US 1991-679075	19910823

PRIORITY APPLN. INFO.: DE 1988-3843930 A 19881224  
WO 1989-EP1548 W 19891215

AB The title monomers, are **prepared** by the **esterification** of (meth)acrylic acid in the presence of acidic **esterification** catalysts and in the presence of  $\alpha$ -tocopherol as a polymerization **inhibitor**. Reaction water is removed from the reaction mixture under elevated temps., producing a reaction mixture without a solvent which would otherwise need to be removed or could form an azeotropic-forming agent. Thus, acrylic acid 324.0, ethoxylated trimethylolpropane (OH value 680 mg KOH/g) 368.2,

p-toluenesulfonic acid 24.2, and D,L- $\alpha$ -tocopherol 1.38 g were heated together at 145° while an air/N mixture (5 volume% O, 40 L/h) was passed through the reaction mixture, the crude product was washed with 700 mL 10% aqueous Na<sub>2</sub>CO<sub>3</sub> solution and dried in vacuum at 80°/40 mbar for 3 h, producing ethoxylated trimethylolpropane acrylates having acid value <1 mg KOH/g, OH value 43 mg KOH/g, Gardner color value 3-4, and H<sub>2</sub>O content 0.31%.

L16 ANSWER 39 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 37

ACCESSION NUMBER: 1990:632240 CAPLUS

DOCUMENT NUMBER: 113:232240

TITLE: **Process** for the manufacture of (meth)acrylate esters of polyhydric **alcohols**

INVENTOR(S): Ritter, Wolfgang; Sitz, Hans Dieter; Speitkamp, Ludwig

PATENT ASSIGNEE(S): Henkel K.-G.a.A., Germany

SOURCE: Ger. Offen., 5 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3843854	A1	19900628	DE 1988-3843854	19881224
EP 376088	A1	19900704	EP 1989-123217	19891215
R: GR				
WO 9007486	A1	19900712	WO 1989-EP1549	19891215
W: JP, US				
RW: AT, BE, CH, DE, ES, FR, GB, IT, LU, NL, SE				
EP 449918	A1	19911009	EP 1990-900856	19891215
EP 449918	B1	19941005		
R: BE, CH, DE, ES, FR, GB, IT, LI, NL				
JP 04502467	T2	19920507	JP 1990-501715	19891215
ES 2061009	T3	19941201	ES 1990-900856	19891215
JP 2935742	B2	19990816	JP 1989-501715	19891215
CA 2006433	AA	19900624	CA 1989-2006433	19891221
US 5350877	A	19940927	US 1991-720443	19910624
PRIORITY APPLN. INFO.:			DE 1988-3843854 A	19881224
			WO 1989-EP1549 W	19891215

AB The title compds. are **prepared** by the reaction of (meth) **acrylic acid** in the presence of **transesterification** catalysts and substituted phenolic polymerization **inhibitors** with polyhydric **alcs.** so as to produce a reaction mixture which is substantially free of solvents and/or azeotropic carriers, and the produced reaction water is removed from the reaction mixture in the gas phase. Thus, acrylic acid 1559.5, ethoxylated trimethylolpropane (I, OH value 680) 1521.0, p-toluenesulfonic acid 107.8, and 2,5-di-tert-butylhydroquinone 4.96 g were contacted with 40 L/h of air to remove water, **esterified** for 6 h, heated at 105°/400 mbar for 6 h, neutralized with 103 g Ca(OH)<sub>2</sub>, stirred at 80°/50 mbar, and filtered to produce I acrylate having acid value <1, OH value 24, and Gardner color >1.

L16 ANSWER 40 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 38

ACCESSION NUMBER: 1991:24763 CAPLUS

DOCUMENT NUMBER: 114:24763

TITLE: **Process** for the manufacture of (meth)acrylate esters of polyhydric **alcohols**

INVENTOR(S): Ritter, Wolfgang; Sitz, Hans Dieter; Speitkamp, Ludwig

10/657,157

PATENT ASSIGNEE(S): Henkel K.-G.a.A., Germany  
 SOURCE: Ger. Offen., 9 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 2  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3843843	A1	19900705	DE 1988-3843843	19881224
EP 376089	A1	19900704	EP 1989-123218	19891215
R: GR				
EP 377156	A1	19900711	EP 1989-123221	19891215
R: GR				
WO 9007483	A1	19900712	WO 1989-EP1546	19891215
W: JP, US				
RW: AT, BE, CH, DE, ES, FR, GB, IT, LU, NL, SE				
WO 9007484	A1	19900712	WO 1989-EP1547	19891215
W: JP, US				
RW: AT, BE, CH, DE, ES, FR, GB, IT, LU, NL, SE				
EP 449865	A1	19911009	EP 1990-900162	19891215
EP 449865	B1	19940928		
R: BE, CH, DE, ES, FR, GB, IT, LI, NL				
EP 449919	A1	19911009	EP 1990-900865	19891215
EP 449919	B1	19940504		
R: BE, CH, DE, ES, FR, GB, IT, LI, NL				
JP 04502455	T2	19920507	JP 1990-500801	19891215
JP 04502459	T2	19920507	JP 1990-501465	19891215
ES 2053171	T3	19940716	ES 1990-900865	19891215
JP 2758717	B2	19980528	JP 1989-501465	19891215
JP 2863309	B2	19990303	JP 1989-500801	19891215
CA 2006430	AA	19900624	CA 1989-2006430	19891221
CA 2006432	AA	19900624	CA 1989-2006432	19891221
AU 618875	B2	19920109	AU 1990-49716	19900212
AU 9049716	A1	19910829		
AU 623222	B2	19920507	AU 1990-49718	19900212
AU 9049718	A1	19910905		
AU 623493	B2	19920514	AU 1990-49722	19900212
AU 9049722	A1	19910829		
AU 626986	B2	19920813	AU 1990-49721	19900212
AU 9049721	A1	19910829		
US 5159106	A	19921027	US 1991-679073	19910820
US 5210281	A	19930511	US 1991-720444	19910823
PRIORITY APPLN. INFO.:			DE 1988-3843843	19881224
			DE 1989-3939163	19891127
			WO 1989-EP1546	19891215
			WO 1989-EP1547	19891215

AB In the title **process**, (meth)acrylic acids are esterified with polyhydric alcs. in the presence of acidic esterification catalysts and liquid droplets of polymerization inhibitor. The crude product may then be neutralized with oxides and/or hydroxides of alkaline earth metals and/or Al, and treated with decoloring agents. Thus, acrylic acid 14.53, ethoxylated trimethylolpropane (OH value 665 mm-KOH/g) 14.18, p-toluenesulfonic acid 1.01, and 2,5-di-tert-butylhydroquinone 0.047 kg were contacted with 100 L/h of air, the product stream heated to 105° and contacted with an air stream (60 L/h), water removed under the following vacuum profile: 2 h at 105°/400 mbar, 1 h at 105°/300 mbar, 0.5 h at 105°/200 mbar, 1 h at 105°/100 mbar, and 0.5 h at

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105°/23 mbar. The intermediate was mixed with 0.53 kg Ca(OH)<sub>2</sub>, stirred at 80°/50 mbars, and filtered, producing polyethylene glycol trimethylolpropane ether acrylate having acid value <1 mg-KOH/g, OH value 14 mg-KOH/g, and Gardner color value <1.

L16 ANSWER 41 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 39

ACCESSION NUMBER: 1990:119592 CAPLUS  
DOCUMENT NUMBER: 112:119592  
TITLE: Manufacture of polyfunctional (meth)acrylate esters with little discoloration  
INVENTOR(S): Honma, Akihiro  
PATENT ASSIGNEE(S): Mitsubishi Gas Chemical Co., Inc., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 01258643	A2	19891016	JP 1988-83104	19880406
JP 2586092	B2	19970226		

PRIORITY APPLN. INFO.: JP 1988-83104 19880406

AB Light-colored transparent title esters are manufactured by **esterifying (meth)acrylic acid** with polyhydric **alcs.** in the presence of an acid catalyst and pyrogallol in contact with O<sub>2</sub>-containing gas, then removing the catalyst by extraction with H<sub>2</sub>O, neutralizing excess **(meth)acrylic acid** with an aqueous alkaline solution, and washing. Thus, a mixture of methacrylic acid 313, trimethylolpropane 148, p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H 34, pyrogallol 0.6, and cyclohexane 130 g was refluxed while bubbling air through the solution, and distilling off 61.5 g H<sub>2</sub>O. Then 242 g cyclohexane and 100 g H<sub>2</sub>O were added, the aqueous layer containing p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H was separated, the organic layer was neutralized by contacting with 372 g 15% aqueous NaOH, then washing with H<sub>2</sub>O, adding 0.01 g toluhydroquinone, and distilling in vacuo to give 350 g trimethylolpropane trimethacrylate with APHA color 20, vs. 60 using hydroquinone instead of pyrogallol.

L16 ANSWER 42 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 40

ACCESSION NUMBER: 1989:407990 CAPLUS  
DOCUMENT NUMBER: 111:7990  
TITLE: Manufacture of esters of unsaturated acids with cation exchangers as catalysts  
INVENTOR(S): Nakajima, Sumio; Sogabe, Hideki; Yoshida, Hiroshi; Okubo, Atsushi  
PATENT ASSIGNEE(S): Nippon Shokubai Kagaku Kogyo Co., Ltd., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 01009956	A2	19890113	JP 1987-162251	19870701

## PRIORITY APPLN. INFO.:

JP 1987-162251

19870701

AB Esters are **prepared** in high yield with min. byproduct formation by**esterification** of (meth)acrylic acidwith C4-12 aliphatic **alcs.** in the presence of strongly acid cation exchange resins as catalysts with stirring at 0.005-2 kW/m<sup>3</sup> in boiling**alc.** solution containing water, the water being removed with **alc**

. and the unreacted acids being recovered in the organic phase. A mixture of

acrylic acid (I) 49, 2-ethylhexanol (II) 36.6, 2-ethylhexyl acrylate 14.2,

and water 0.1% was added to a reactor containing 14 L ion exchange resin

(Diaion PK-208) to total volume 50 L, and the composition was stirred at

85°/70 mm with continuous addition of the mixture at 23.5 kg/h and II at

13.2 kg/h, addition of polymerization **inhibitor**, and removal of 1.69 kg/h

aqueous phase containing 0.002% I and 0.08% II and 0.18 kg/h organic phase

containing I

0.003, isooctane 3.7, and H<sub>2</sub>O 2.5%, the conversions of I and II being

59.5% and 56.8%, resp., and the selectivities for I and II being 99.2% and

99.12%, resp.

L16 ANSWER 43 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 41

ACCESSION NUMBER: 1989:407985 CAPLUS

DOCUMENT NUMBER: 111:7985

TITLE: **Preparation** of (dialkylamino)alkyl  
(meth)acrylatesINVENTOR(S): Hurtel, Patrice; Hazan, Charles; De Champs, Francois;  
Paul, Jean Michel

PATENT ASSIGNEE(S): Norsolor S. A., Fr.

SOURCE: Eur. Pat. Appl., 4 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 298867	A2	19890111	EP 1988-401771	19880707
EP 298867	A3	19890308		
EP 298867	B1	19920923		
R: AT, BE, CH, DE, ES, GB, GR, IT, LI, LU, NL, SE				
FR 2617840	A1	19890113	FR 1987-9697	19870708
FR 2617840	B1	19890901		
CA 1303056	A1	19920609	CA 1988-571353	19880707
AT 80868	E	19921015	AT 1988-401771	19880707
ES 2034317	T3	19930401	ES 1988-401771	19880707
JP 01038047	A2	19890208	JP 1988-170723	19880708
US 4851568	A	19890725	US 1988-216593	19880708

PRIORITY APPLN. INFO.:

FR 1987-9697

19870708

EP 1988-401771

19880707

OTHER SOURCE(S): CASREACT 111:7985; MARPAT 111:7985

AB The esters CH<sub>2</sub>:C(R<sub>1</sub>)CO<sub>2</sub>ZNR<sub>2</sub>R<sub>3</sub> (R<sub>1</sub> =H, Me; R<sub>2</sub>, R<sub>3</sub> =alkyl, aryl, or form a ring; Z =C<sub>1</sub>-5 alkylene) are **prepared** by heating Et (meth)acrylate with R<sub>3</sub>R<sub>2</sub>NZOH in the presence of Ti(OEt)<sub>4</sub> and polymerization **inhibitors**. Thus, heating 660 parts Et acrylate, 267 parts Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>OH, 1000 ppm phenothiazine, and 0.5 mol% (based on amino **alc.**) Ti(OEt)<sub>4</sub> at 90-95° and 0.53 bar with distillation of acrylate-EtOH azeotrope gave 422 parts 2-(dimethylamino)ethyl acrylate.

L16 ANSWER 44 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 42

ACCESSION NUMBER: 1989:515916 CAPLUS

DOCUMENT NUMBER: 111:115916



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TITLE: Preparation of (meth)  
acrylic acid esters by  
transesterification  
INVENTOR(S): Beranek, Jan; Gutwirth, Karel; Machova, Marta; Benes,  
Radek; Kantor, Milan  
PATENT ASSIGNEE(S): Czech.  
SOURCE: Czech., 5 pp.  
CODEN: CZXXA9  
DOCUMENT TYPE: Patent  
LANGUAGE: Czech  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 259632	B1	19881014	CS 1987-1241	19870225
PRIORITY APPLN. INFO.:			CS 1987-1241	19870225
OTHER SOURCE(S):		CASREACT 111:115916		

AB The **transesterification** of CH<sub>2</sub>:CRCO<sub>2</sub>R<sub>1</sub> (R = H, Me, R<sub>1</sub> = Me, Et) with higher aliphatic or cycloaliph. **alcs.** and glycols is catalyzed by Mg(OMe)<sub>2</sub>, which is easy to remove as MgCO<sub>3</sub>. Thus, a mixture of Alfol 1620 (cetyl and stearyl **alc.**) 434, CH<sub>2</sub>:CMeCO<sub>2</sub>Me (I) 194, cyclohexane 333, and phenyl-β-naphthylamine (polymerization **inhibitor**) 1 g was azeotropically dehydrated; a suspension of 0.7 g Mg in 14 g MeOH was added at 70°; and the mixture was refluxed 4 h with separation of MeOH. The residual solution was diluted with 4 g water and treated 15 min with 172 mL/min CO<sub>2</sub>, which was heated to 95° in a hot washing bottle. Residual CO<sub>2</sub> was flushed with air, precipitated MgCO<sub>3</sub> was filtered with kieselguhr, and solvents and I were steam-stripped to give 98% cetyl-stearyl methacrylate containing <2% alcoholates and <0.5% I. The filter cake containing 70% MgCO<sub>3</sub> was utilized as a fertilizer component.

L16 ANSWER 45 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 43

ACCESSION NUMBER: 1988:205252 CAPLUS  
DOCUMENT NUMBER: 108:205252  
TITLE: **Process** for producing unsaturated  
carboxylates  
INVENTOR(S): Nakashima, Sumio; Sogabe, Hideki; Yoshida, Hiroshi;  
Okubo, Atsushi  
PATENT ASSIGNEE(S): Nippon Shokubai Kagaku Kogyo Co., Ltd., Japan  
SOURCE: PCT Int. Appl., 25 pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 8800180	A1	19880114	WO 1987-JP482	19870707
W: BR, HU, KR, US				
RW: DE, FR, GB, IT				
JP 63017844	A2	19880125	JP 1986-159674	19860709
EP 273060	A1	19880706	EP 1987-904331	19870707
EP 273060	B1	19911016		
R: DE, FR, GB, IT				
BR 8707412	A	19881101	BR 1987-7412	19870707
HU 48863	A2	19890728	HU 1987-3812	19870707
HU 204024	B	19911128		
CA 1290767	A1	19911015	CA 1987-541491	19870707

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CN 87105726	A	19880706	CN 1987-105726	19870709
CN 1024190	B	19940413		
CS 272223	B2	19910115	CS 1987-5239	19870709
US 4833267	A	19890523	US 1988-193523	19880309
PRIORITY APPLN. INFO.:			JP 1986-159674	19860709
			WO 1987-JP482	19870707

OTHER SOURCE(S): CASREACT 108:205252

AB In **esterification** of (meth)acrylic acid with C1-12 aliphatic **alcs.**, a strongly acidic ion exchange resin catalyst is dispersed in the reaction mixture by a mixer having a stirring power of 0.005-2 kW/m<sup>3</sup> reaction solution. Thus, 36.7 kg/h mixture solution containing acrylic acid 31.4, 2-ethylhexanol 59.4,

2-ethylhexyl

acrylate (I) 9.1, and H<sub>2</sub>O 0.1% was fed to a reactor containing 14 L Diaion PK 208 and 0.05% polymerization **inhibitor**, and the reaction mixture was stirred (0.05 kW/m<sup>3</sup>) at 85°/70 mmHg to give a bottom discharge containing 59.9% I with 58.9% conversion (based on acrylic acid) and 99.19% selectivity.

L16 ANSWER 46 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 44

ACCESSION NUMBER: 1988:427631 CAPLUS

DOCUMENT NUMBER: 109:27631

TITLE: (Meth)acrylate dental material

INVENTOR(S): Schmitt, Werner; Jochum, Peter; Zahler, Wolf Dietrich; Huebner, Heijo; Holupirek, Manfred; Gasser, Oswald; Herzig, Christian

PATENT ASSIGNEE(S): ESPE Fabrik Pharmazeutischer Praeparate G.m.b.H., Fed. Rep. Ger.

SOURCE: Ger. Offen., 5 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3607331	A1	19870910	DE 1986-3607331	19860306
US 4795823	A	19890103	US 1987-20902	19870302
JP 62230750	A2	19871009	JP 1987-49874	19870303
JP 07068172	B4	19950726		
EP 235826	A1	19870909	EP 1987-103200	19870306
EP 235826	B1	19900425		
R: AT, BE, CH, DE, FR, GB, LI, LU, NL, SE				
AT 52245	E	19900515	AT 1987-103200	19870306
PRIORITY APPLN. INFO.:			DE 1986-3607331	19860306
			EP 1987-103200	19870306

AB The (meth)acrylic acid esters

(MO)<sub>n</sub>(AO<sub>2</sub>CCH<sub>2</sub>OCH<sub>2</sub>CH<sub>2</sub>OCH<sub>2</sub>CO<sub>2</sub>)<sub>x</sub>A(OM)<sub>n</sub> [A = **alc.** radical with C<sub>≥4</sub> between the linkage sites; M = CH<sub>2</sub>:CR<sub>1</sub>CO; R<sub>1</sub> = H, Me; n = 1, 2; x = 0.3-3] are **prepared** as dental material.

Bis(hydroxymethyl)tricyclo[5.2.1.0<sup>2,6</sup>]decane (T-diol) (196 g) in 400 mL cyclohexane was partially **esterified** with 89 g triglycolic acid in the presence of 7 g 4-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H with the removal of 18 g H<sub>2</sub>O. The free OH groups were **esterified** with 129 g CH<sub>2</sub>:CMeCO<sub>2</sub>H in the presence of polymerization **inhibitors** to give an ester mixture consisting of 32% T-diol dimethacrylate, 38% triglycolic acid bis[T-methacrylate] and 30% HO(TOCHOCH<sub>2</sub>CH<sub>2</sub>OCH<sub>2</sub>CO<sub>2</sub>)<sub>2</sub>-3TOH bis(methacrylate). A solution was **prepared** of 50 parts by weight bis(acryloyloxymethyl)tricyclo[5.2.1.0<sup>2,6</sup>]decane and 50 parts of the ester mixture and 0.15% camphorquinone and 1.5%

N,N-dimethylaminoethyl methacrylate added. Sep., a powder was **prepared** containing 112 parts silanized SiO<sub>2</sub> granulate, 72 parts silanized pyrogenic SiO<sub>2</sub> and 3 g CaF<sub>2</sub>. A dental composition was **prepd** by mixing 113 g of the powder with 80 mL of the solution Polymerization was carried out by light exposure as usual.

L16 ANSWER 47 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 45  
 ACCESSION NUMBER: 1987:157005 CAPLUS  
 DOCUMENT NUMBER: 106:157005  
 TITLE: (Meth)acrylate esters  
 INVENTOR(S): Fujii, Masahiko; Hashino, Shizuo  
 PATENT ASSIGNEE(S): Nippon Oil Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 61243046	A2	19861029	JP 1985-82267	19850419
PRIORITY APPLN. INFO.:			JP 1985-82267	19850419
OTHER SOURCE(S): CASREACT 106:157005				
AB (Meth)acrylate esters, useful as monomers for resins, coating materials, and adhesives, are <b>prepared by esterification of alcs.</b> with excess (meth)acrylic acid in the presence of acid catalysts and polymerization <b>inhibitors</b> . The reaction mixture is neutralized with aqueous alkali solns., inorg. acids are added to pH ≤4, and the unesterified acid is extracted with aliphatic or petroleum ethers from the aqueous layer. CH <sub>2</sub> :CMeCO <sub>2</sub> H was treated with Me(CH <sub>2</sub> ) <sub>17</sub> OH in the presence of 4-MeC <sub>6</sub> H <sub>4</sub> SO <sub>3</sub> H and hydroquinone at 110-115°/200-300 torr for 4.5 h, then the solution was neutralized with 17.5% aqueous NaOH, and separated from the oil layer to give 95.8% CH <sub>2</sub> :CMeCO <sub>2</sub> (CH <sub>2</sub> ) <sub>17</sub> Me. The aqueous layer was extracted with Et <sub>2</sub> O to recover the unesterified starting acid in 99.5% purity and reduce the COD of the effluent by 79.5%.				

L16 ANSWER 48 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 46  
 ACCESSION NUMBER: 1987:67824 CAPLUS  
 DOCUMENT NUMBER: 106:67824  
 TITLE: (Meth)acrylate esters  
 INVENTOR(S): Hashino, Shizuo; Tanabe, Tatsuhei  
 PATENT ASSIGNEE(S): Nippon Oils & Fats Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 61176555	A2	19860808	JP 1985-14447	19850130
JP 06080033	B4	19941012		
PRIORITY APPLN. INFO.:			JP 1985-14447	19850130
AB (Meth)acrylate esters are <b>prepared by esterifying higher alcs.</b> with excess (meth)acrylic acid in presence of both an <b>esterifying</b> catalyst and a polymerization <b>inhibitor</b> , followed by <b>esterifying</b> residual (				

**meth)acrylic acid** with lower **alcs.**, and fractionating the mixed (meth)acrylate esters. This method gives higher **alc.** esters in high yield and purity with efficient recovery of the excess acid. Thus, heating H<sub>2</sub>C:CM<sub>2</sub>CO<sub>2</sub>H (I) 103.3, octadecyl **alc.** 270.0, 4-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H 3.5, and hydroquinone 0.11 g at 110-115°/200-600 torr for 4.5 h gave a 99.2% conversion of the **alc.** and 13.6 g residual I. Heating the mixture with 30.3 g MeOH at 80° for 3 h and distilling the final mixture at 46°/160 torr gave a distillate containing 15.1 g Me methacrylate corresponding to a 95.2% recovery of residual I, and a residue containing 324.6 g octadecyl methacrylate corresponding to a 96.0% recovery of the **alc.**

L16 ANSWER 49 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 47  
 ACCESSION NUMBER: 1987:120368 CAPLUS  
 DOCUMENT NUMBER: 106:120368  
 TITLE: Alkyl methacrylate **preparation**  
 INVENTOR(S): Dietrich, Gerhard; Nestler, Gerhard; Ruckh, Peter; Herzog, Reinhard  
 PATENT ASSIGNEE(S): BASF A.-G., Fed. Rep. Ger.  
 SOURCE: Ger. Offen., 8 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3518482	A1	19861127	DE 1985-3518482	19850523
US 4675436	A	19870623	US 1986-857052	19860429
JP 61271247	A2	19861201	JP 1986-108733	19860514
JP 06017337	B4	19940309		
EP 202610	A2	19861126	EP 1986-106633	19860515
EP 202610	A3	19870128		
EP 202610	B1	19880928		

R: BE, DE, FR, GB, IT, NL

PRIORITY APPLN. INFO.: DE 1985-3518482 19850523

OTHER SOURCE(S): CASREACT 106:120368

AB Alkyl (meth)acrylates are **prepared** from (meth)**acrylic acid** and C<sub>6</sub>-20 **alcs.** in the presence of 0.1-5% strong acids, polymerization **inhibitors**, and an O-containing gas (1-20 vol%) at 80-150°, for the elimination of H<sub>2</sub>O. The (meth)**acrylic acid-alc.** ratio is 1:0.8-1.2. To a 1st reactor was fed methacrylic acid 190, H<sub>2</sub>SO<sub>4</sub> 5, phenothiazine 0.3, and n-octanol 280 parts/h. To a 2nd reactor was fed the mixture from the 1st reactor with 36 parts/h n-octanol, and 150,000 volume parts air. At 120°, 40 parts/h H<sub>2</sub>O was eliminated. The product (470 parts/h) contained n-octyl methacrylate 90.5, methacrylic acid 0.7, octanol 5.8, and high-boiling side products (traces of dioctyl ether and olefins) 0.5%.

L16 ANSWER 50 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 48  
 ACCESSION NUMBER: 1984:121755 CAPLUS  
 DOCUMENT NUMBER: 100:121755  
 TITLE: Acrylate or methacrylate esters  
 PATENT ASSIGNEE(S): Yokkaichi Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese

10/657,157

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 58174346	A2	19831013	JP 1982-57285	19820408
JP 01042255	B4	19890911		

PRIORITY APPLN. INFO.: JP 1982-57285 19820408

AB Acrylic acid (I) [79-10-7] or methacrylic acid [79-41-4] is **esterified** with **alcs.** in the presence of **esterification** catalysts, antioxidants, and Cu or Cu salts (polymerization **inhibitors**), and the products are treated with pyrophosphoric acid (II), II salts, 1-hydroxyethylidenebis(phosphonic acid) (III) [2809-21-4], or III salts to remove Cu ions. Thus, a mixture of 1.10 equiv I, 1.0 equiv 1,1'-isopropylidenebis[4-[2-(2-hydroxethoxy)ethoxy]benzene] [27697-57-0], 3.4% (based on I) 4-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H, 100 ppm (on I) powdered Cu, 100° (on I) PhMe, and 0.4% NaH<sub>2</sub>PO<sub>2</sub> was heated to reflux, removing H<sub>2</sub>O until the acid number reached a constant value and the product cooled, neutralized with dilute NaOH, treated with 0.55% (on I) III, separated from the aqueous phase, and stripped of solvents in vacuo at 50-60° to give an ester [56361-55-8] having APHA color number 30 and containing only a trace of Cu ion.

L16 ANSWER 51 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 49

ACCESSION NUMBER: 1981:192923 CAPLUS

DOCUMENT NUMBER: 94:192923

TITLE: Monomers for polymeric depressing additives for high-paraffin oils

INVENTOR(S): Minkov, V. A.; Sopina, V. E.; Zakordonets, O. P.; Sergeev, V. P.; Gaevoi, G. M.; Khmel'nitskii, A. G.; Lubenets, E. G.; Saveleva, N. I.; Skovorodnikov, Yu. A.

PATENT ASSIGNEE(S): USSR

SOURCE: U.S.S.R. From: Otkrytiya, Izobret., Prom. Obraztsy, Tovarnye Znaki 1981, (9), 88.

CODEN: URXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Russian

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
SU 810668	A1	19810307	SU 1978-2694189	19781206

PRIORITY APPLN. INFO.: SU 1978-2694189 19781206

AB Monomers for pour-point-depressant polymers were **prepared** by simultaneous **esterification** of acrylic and methacrylic acid, in a 2-5:1 mol. ratio, with a C<sub>18</sub>-26 **alc.** fraction in toluene. The reaction was carried out at 100-20° using toluenesulfonic acid [104-15-4] as catalyst and hydroquinone [123-31-9] as polymerization **inhibitor**.

L16 ANSWER 52 OF 52 CAPLUS COPYRIGHT 2004 ACS on STN DUPLICATE 50

ACCESSION NUMBER: 1981:47993 CAPLUS

DOCUMENT NUMBER: 94:47993

TITLE: (Meth)acrylic acid esters

INVENTOR(S): Schuster, Karl Ernst; Rosenkranz, Hans Juergen; Griehsel, Bernd

10/657,157

PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Ger. Offen., 19 pp.  
CODEN: GWXXBX  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2913218	A1	19801023	DE 1979-2913218	19790403

PRIORITY APPLN. INFO.: DE 1979-2913218 19790403

AB (Meth)acrylates of saturated aliphatic **alcs.** containing 2-4 OH groups, their ethoxylation products, or unsubstituted or C1-3 alkyl mono- or disubstituted 5-(hydroxymethyl)-1,3-dioxanes are **prepared** by **esterification** in the presence of 0.001-5% organic phosphite catalyst and 0.01-0.3% mono- or diphenol as polymerization **inhibitor**. Thus, acrylic acid [79-10-7] 3.5, ethoxylated trimethylolpropane [50586-59-9] 5.36, cyclohexane 2.7, and H2SO4 0.073 kg were mixed with 0.006 kg (EtO)3P [122-52-1] and 2,5-di-tert-butylhydroquinone (I) [88-58-4] and **esterified** 17 h at 82°, while 10 L/h (EtO)3P-saturated air and 10 L/h I-saturated air were passed through the mixture, so that the acid number of the reaction mixture was 12. The mixture was cooled, mixed with (EtO)3P 0.005, I 0.0024, and toluhydroquinone [95-71-6] 0.002 kg, distilled free of cyclohexane, and distilled for 4 h at 50 mbar/105° while 50 L/h (EtO)3P-saturated air was passed through the mixture. The final product [28961-43-5] had acid number 2.5, iodine color number 0-1, and viscosity 120 mPa-s. A control **prepared** without (EtO)3P had iodine color number 2-3 and viscosity 190 mPa-s.

=> log y		
COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	195.12	195.33
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	-38.22	-38.22

STN INTERNATIONAL LOGOFF AT 13:39:59 ON 28 JUL 2004